

Claims

1. A column for use in an analytical temperature rising elution fractionation analysis of a crystalline or a semi-crystalline polymer sample solution characterized in that the packing of said column comprises elastic wires.
5
2. Column according to claim 1, wherein the packing of said column comprises elastic wires, having a Young modulus higher than 50 GPa.
3. Column according to any of claims 1 or 2, wherein the packing of said column comprises
10 elastic wires having a thermal conductivity higher than $0.1 \text{ W cm}^{-1} \text{ K}^{-1}$.
4. Column according to any of claims 1 to 3, wherein the packing of said column comprises elastic wires having a length of at least 2mm and a diameter of less than 1mm.
15
5. Column according to any of claims 1 to 4, wherein said elastic wires are made of stainless steel, metal, carbon fibers or glass fibers.
6. Column according to any of claims 1 to 5, wherein the packing of said column comprises elastic wires having a length per diameter (L/D) of at least 3.
20
7. Column according to any of claims 1 to 6, wherein said column is made of stainless steel, glass, ceramic, or a polymer.
8. Column according to any of claims 1 to 7, said column having an internal diameter less
25 than 20 mm a length comprised between 50 and 500 mm.
9. Device for use in an analytical temperature rising elution fractionation analysis comprising the column according to any of claims 1 to 8, a temperature controlling system for controlling the temperature of said column, a sample injector for injecting a polymer sample solution into
30 said column, a pump for eluting the polymer fractions from said column and a detector for detecting eluting fractions of said sample solution.

10. Device according to claim 9, wherein said detector is a differential refractive index (DRI) detector.

11. A method for performing an ATREF analysis of a crystalline or a semi-crystalline polymer

5 solution comprising the steps of:

- injecting a sample of said polymer solution into the column according to any of claims 1 to 8,
- crystallizing said polymer sample solution in said column over a cooling temperature gradient to produce a crystallized polymer sample while keeping solvent flowing 10 through said column;
- eluting said crystallized polymer sample by increasing the temperature of said column over a heating temperature gradient to produce eluted fractions of the polymer sample solution, said fractions being eluted in function of the temperature; and
- measuring the concentration of the eluted fractions of polymer sample solution by 15 means of a detector.

12. Method according to claim 11, wherein the concentration of the eluted fractions of polymer sample solution are measured using a differential refractive index detector.

20 13. Method according to claim 11 or 12, wherein the column is provided in a temperature controlling system.

14. Method according to any of claims 11 to 13, wherein an amount of polymer lower than 2.0 mg polymer is injected into the column.

25 15 Method according to any of claims 11 to 14, wherein a volume of the polymer sample solution lower than 1.0 ml is injected into the column.

16. Method according to any of claims 11 to 15, wherein said polymer sample solution is 30 injected automatically.

17. Method according to claim 16, wherein said polymer sample solution is injected automatically at a flow rate lower than 2.0 mL/min.

18. Method according to any of claims 11 to 17, wherein the crystallization of said polymer sample solution in said column is performed over a cooling temperature gradient with a maximum temperature below 210°C.

5

19. A method according to any of claims 11 to 18, wherein the crystallization of said polymer sample solution in said column is performed at a cooling rate higher than 0.5 °C per minute.

10 20. Method according to any of claims 11 to 19, wherein the elution of the crystallized polymer sample is performed over a heating temperature gradient with a maximum temperature up to 210°C.

21. Method according to any of claims 11 to 20, wherein the elution of the crystallized polymer sample is performed at a heating rate higher than 0.5°C per minute.

15